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TITLE: Process for the preparation of acylated hexaazaisowurtzitane derivatives

Brief Summary Text (196):

With respect to the synthesis of a hexaacylhexaazaisowurtzitane, preferred examples of acylating agents usable therefor include acyl halides, such as acetyl chloride, acetyl bromide and the like.

Brief Summary Text (208):

The membrane filtration separation method, which is a method for removing the first solvent from the composition system to deposit crystals of the desired compound, employs a separation membrane having a selective permeability for the first solvent. When the removal of the first solvent is performed by using the membrane separation method, it is most preferred that the first solvent is water. The type of the separation membrane to be used for separating water from the composition system can be selected in accordance with various factors, such as the shape of the reactor and the like. As examples of separation membranes, there can be mentioned conventional separation membranes for separating water from ethanol. Specific examples of such separation membranes include a polyhydroxymethylene membrane; an acrylic acid-acrylonitrile copolymer membrane; an ionized chitosan membrane; organic membranes, such as a composite membrane having an active layer of maleic acid-crosslinked PVA, and a polymer alloy membrane (see Unexamined Japanese Patent Application Laid-Open Specification Nos. 59-109204 and 60-129104); and an inorganic membrane, such as an Azeolite membrane described in "Kagakukougaku Sinpojyumu Siriizu (Chemical Engineering Symposium Series)" 41, p. 102-105 (1994). From the viewpoint of the commercial practice of the method of the present invention by using the membrane filtration separation method, it is preferred to use a membrane having an excellent durability, high resistance against the chemicals contained in a reaction system and a reaction mixture, and high mechanical strength. An example of such a preferred membrane is an inorganic membrane since it generally has a high operation limit temperature and high chemical resistance. The above-mentioned A-zeolite membrane can be prepared in accordance with a method described in "Kagakukougaku Sinpojyumu Siriizu (Chemical Engineering Symposium Series)" 41, p. 102-105 (1994), that is, a method in which a porous alumina substrate having a pore diameter of approximately 1 mm is immersed in a solution containing sodium silicate, sodium hydroxide, sodium aluminate and aluminum hydroxide in weight ratios of H_{sub}2 O/Na_{sub}2 O=60, Na_{sub}2 O/SiO_{sub}2 =1, and SiO_{sub}2 /Al_{sub}2 O_{sub}3 =2, and then subjected to hydrothermal reaction at 80 to 100 degree C. for 3 to 12 hours. The properties of the membrane can be controlled by repeating the above-mentioned immersion and hydrothermal reaction.

Detailed Description Text (120):

Substantially the same reaction as described in Example 5 was repeated except that diethylene glycol diethyl ether was used as the reaction solvent. The resultant reaction mixture was taken out from the autoclave and the catalyst contained in the reaction mixture was filtered off to thereby obtain a solution as a filtrate. 2 g of acetyl chloride was added to the solution and stirred at room temperature for 1 hour to perform a reaction. By this reaction, the N-H groups of tetraacetylhexaazaisowurtzitane were acylated, so that hexaacetylhexaazaisowurtzitane was obtained in an approximately stoichiometric amount, based on the tetraacetylbenzylhexaazaisowurtzitane. The resultant reaction

mixture was subjected to distillative crystal deposition in substantially the same manner as described in Example 3, to thereby obtain crystals of hexaacetylhexaazaisowurtzitane in a yield of 90%, based on the tetraacetylbenzylhexaazaisowurtzitane [crystal deposition yield: 85%, purity: 97% (determined by HPLC)].

Detailed Description Text (123):

Substantially the same procedure as described in Example 3 was repeated except that, as the reaction solvent for the first-stage reaction, diethylene glycol dimethyl ether was used, that the reaction time for the first-stage reaction was changed to 10 hours, and that, as a first solvent which was added to the reaction system at a point in time between the first-stage reaction and the second-stage reaction, acetic acid was used. The resultant reaction mixture was taken out from the autoclave and the catalyst contained in the reaction mixture was filtered off to thereby obtain a solution as a filtrate. 2.0 g of acetyl chloride was added to the solution and stirred at 40. degree. C. for 1 hour. The resultant reaction mixture was subjected to distillative crystal deposition in substantially the same manner as described in Example 3, to thereby obtain crystals of hexaacetylhexaazaisowurtzitane in a yield of 64%, based on the hexabenzylhexaazaisowurtzitane [crystal deposition yield: 85%; purity: 98% (determined by HPLC)].